

# Sertifikaat

PATENTKANTOOR

1B 99 / 1747

DEPARTEMENT VAN HANDEL  
EN NYWERHEID

REPUBLIEK VAN SUID-AFRIKA



REPUBLIC OF SOUTH AFRICA

REC'D 14 DEC 1999

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PATENT OFFICE

DEPARTMENT OF TRADE  
AND INDUSTRY

# Certificate

Hiermee word gesertifiseer dat  
This is to certify that

the attached documents attached hereto are true copies of the Forms P2, P6,  
provisional specification and drawings of South African Patent Application No. 98/9867 in  
the name of BRITS, Willem Hendrik (assigned from IMMALYTICA (PROPRIETARY)  
LIMITED, formerly ISLANDSITE INVESTMENTS ONE (PTY) LTD)

Filed : 29.10.98

Entitled : ASSAY FLUXES

## PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN  
COMPLIANCE WITH RULE 17.1(a) OR (b)

kente  
dat PRETORIA

in die Republiek van Suid-Afrika, hierdie  
in the Republic of South Africa, this

12th dag van  
day of

November 1999

Registrateur van Patente  
Registrar of Patents

REPUBLIC OF SOUTH AFRICA			REGISTER OF PATENTS			PATENTS ACT, 1	
OFFICIAL APPLICATION			LODGING DATE: PROVISIONAL			ACCEPTANCE DATE	
21	01	989867	22	29 OCTOBER 1998		47	
INTERNATIONAL CLASSIFICATION			LODGING DATE: COMPLETE			GRANTED DATE	
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FULL NAME(S) OF APPLICANT(S)/PATENTEE(S)							
71	ISLANDSITE INVESTMENTS ONE (PTY) LTD Name changed: Immalytica Pty Ltd. 27-1-99 LANSGEWERKERS VEEVANG						
APPLICANTS SUBSTITUTED			APPLICANTS SUBSTITUTED			DATE REGISTERED	
71	Willem Hendrik Brits					22-10-99	
ASSIGNEE(S)						DATE REGISTERED	
71							
FULL NAME(S) OF INVENTOR(S)							
72	WILLEM HENDRIK BRITS						
PRIORITY CLAIMED		COUNTRY		NUMBER		DATE	
N.B. Use International abbreviation for country (see Schedule 4)		33	NONE		31	NONE	
TITLE OF INVENTION							
54	ASSAY FLUXES						
ADDRESS OF APPLICANT(S)/PATENTEE(S)							
29 BOEKENHOUT STREET, DELPARK, EXT 5, BRAKPAN, SOUTH AFRICA							
ADDRESS FOR SERVICE						S AND F REF	
74	SPOOR AND FISHER, SANDTON				JP/I 175/DC/acm		
PATENT OF ADDITION NO.			DATE OF ANY CHANGE				
61							
FRESH APPLICATION BASED ON			DATE OF ANY CHANGE				

REPUBLIC OF SOUTH AFRICA  
PATENTS ACT, 1978  
APPLICATION FOR A PATENT  
AND ACKNOWLEDGEMENT OF RECEIPT  
(Section 30 (1) - Regulation 22)

REPUBLIC OF SOUTH AFRICA FORM P-1  
REVENUE

29.10.93

R 060.00

The granting of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate

IN KOMSTE  
REPUBLIEK VAN SUID AFRIKA  
S AND F REFERENCE

## OFFICIAL APPLICATION NO.

21

01

989867

JP/I 175/DC/acm

## FULL NAME(S) OF APPLICANT(S)

71

ISLANDSITE INVESTMENTS ONE (PTY) LTD - Name changed 27-1-94

Immatrica Pty Ltd

AANSOEKERSVERVANG (J)  
APPLICANTS SUBSTITUTED

William Hendrik Brits 22-10-94

ADDRESS(ES) OF APPLICANT(S)

29 BOEKENHOUT STREET, DELPARK, EXT 5, BRAK PAN, SOUTH AFRICA

## TITLE OF INVENTION

54

ASSAY FLUXES

THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. THE EARLIEST PRIORITY CLAIM IS:

COUNTRY: NONE

NUMBER: NONE

DATE: NONE

THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.

21	01	
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THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND IS BASED ON APPLICATION NO.

21	01	
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## THIS APPLICATION IS ACCCOMPANIED BY:

- 1. A single copy of a provisional or two copies of a complete specification of 8 pages.
- 2. Drawings of 1 sheets.
- 3. Publication particulars and abstract (Form P.8 in duplicate).
- 4. A copy of Figure of the drawings (if any) for the abstract.
- 5. An assignment of invention.
- 6. Certified priority document(s).
- 7. Translation of the priority document(s).
- 8. An assignment of priority rights.
- 9. A copy of the Form P.2 and the specification of S.A. Patent Application No.
- 10. A declaration and power of attorney on Form P.3.
- 11. Request for ante-dating on Form P.4.
- 12. Request for classification on Form P.9.
- 13. Form P.2 in duplicate.

74 ADDRESS FOR SERVICE: SPOOR AND FISHER, SANDTON

Dated: 29 OCTOBER 1998

*mf lemoen*

SPOOR AND FISHER  
PATENT ATTORNEYS FOR THE APPLICANT(S)

REGISTRAR OF PATENTS, TRADE MARKS AND COPYRIGHT
1993-10-29
FREDORIA
REGISTRATEUR VAN PATENTE, MERKEN MANUELSMERKEN EN DOUTEURAFSKEDE
REGISTRAR OF PATENTS

REPUBLIC OF SOUTH AFRICA  
PATENTS ACT, 1978

## PROVISIONAL SPECIFICATION

(Section 30(1) – Regulation 27)

OFFICIAL APPLICATION NO.

LODGING DATE

21	01	989867	22	29 OCTOBER 1998
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FULL NAME(S) OF APPLICANT(S)

71	ISLANDSITE INVESTMENTS ONE (PTY) LTD ImmaLytica (Pty) Ltd -	Name changed 27-1-99
	AANGEKENDE VERVANG APPLICANTS SUBSTITUTED	Willem Hendrik Brits. (22-10-99.)

FULL NAME(S) OF INVENTOR(S)

72	WILLEM HENDRIK BRITS
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TITLE OF INVENTION

54	ASSAY FLUXES
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989867

#### BACKGROUND OF THE INVENTION

This invention relates to assay fluxes.

In order to plan and manage mining operations and to estimate recoverable ore reserves it is necessary to have the facility to determine the concentration in ore samples of precious metals, typically gold and PGMs (platinum group metals including platinum, palladium, rhodium, osmium,

indium and ruthenium). It is already known to use a Fire Assay process for this purpose. In Fire Assay, PGMs and gold are separated from gangue materials by collection into either lead or nickel sulphide at temperatures of around 1200 - 1450°C. This is achieved by mixing an aliquot of the sample with a flux containing either lead oxide, for the lead collection, or a combination of nickel carbonate and sulphur, for the nickel sulphide, with other chemicals. This mixture is placed into a ceramic crucible, which in turn is placed into an electric or gas fired furnace and heated to an appropriate temperature for a period of about 90 minutes. During this time the mixture melts and, because their chemical affinity, PGMs and gold are collected into either lead or nickel sulphide. On cooling the lead or nickel sulphide is separated from the gangue material and the PGMs and gold content determined by a number of analytical techniques.

A flux containing lead oxide which has been found to work satisfactorily in the conventional fire assay method consists of calcium carbonate, lead oxide, borax and silica, and a carbon source such as activated carbon, maize meal or flour.

In a recent development it has been proposed to use an induction furnace in the fire assay method, as described in a co-pending provisional patent application in the name of the applicant of this application entitled "assaying", which is incorporated herein by reference. Although the fire assay procedure is far quicker than with conventional electrical resistance heating, it has been found that the sample can blow out, spatter and boil over from the crucible as it is heated to melting. This is attributable to the rapid heating and release of air trapped in the sample/flux powder, and to the release of CO and CO<sub>2</sub>, and results in unacceptable sample losses and poor assay results.

## **SUMMARY OF THE INVENTION**

According to the invention there is provided a flux composition for use in fire assaying of ore samples, the composition containing sodium hydroxide.

Preferably, the flux composition comprises 20% to 60%, more preferably 25% to 40%, by weight, sodium hydroxide.

The composition may further comprise:

20% to 50%, preferably 30% to 40%, by weight, lead oxide; and  
30% to 50%, preferably 20% to 60%, by weight, borax.

Advantageously, the composition may also comprise 0 to 1% silver nitrate.

According to a further aspect of the invention, there is provided a sealed container, made from a carbon-based material, containing a predetermined amount and composition of flux as described above.

Typically, the container is made from a plastics material.

## **BRIEF DESCRIPTION OF THE DRAWINGS**

The invention will now be described in more detail, by way of example only, with reference to the accompanying drawings.

Figure 1 is a schematic flow diagram of a process for assaying an ore sample to determine the concentration of selected metals therein, using a flux composition according to the invention;

Figure 2 is a pictorial view of a bottle containing flux according to the invention; and apparatus according to the invention for carrying out the process of Figure 1; and

Figure 3 is a cross-sectional view of an induction furnace in which a flux composition according to the invention is heated.

#### **DESCRIPTION OF AN EMBODIMENT**

Referring to Figure 1, a flux according to the invention is provided for use in a method of assaying an ore sample, the method including the steps of preparing an ore sample 10, mixing the ore sample 10 with the flux 12 which includes lead oxide, to form a mixture 14, inductively heating the mixture 14 to form a fusion of slag 16 and lead 18 containing gold and PGMs, separating the lead 18 from the slag 16 and determining the amount of gold and PGMs in the lead.

In the sample preparation step, an ore sample is logged and its bulk weight determined. The ore sample is then dried, comminuted and sieved and split at into a number of aliquots which are deposited into storage containers in the form of small plastic bottles, each of which already contains a flux. The unused portion of the ore sample is sealed in a storage container and identified by means of a bar code label indicating identification, origin and date of assay.

A typical bottle 20 is shown in Figure 2. The bottle 20 is made from a plastics material (polyethylene) and has a lid 22 which engages sealingly with the opening into the bottle 20. A charge of flux 26 according to the invention is shown in the bottle 20.

The flux 26 comprises:

20% to 60%, preferably 25% to 40%, by weight, sodium hydroxide (NaOH);

20% to 50%, preferably 30% to 40%, by weight, lead oxide;

20% to 50%, preferably 30% to 40%, by weight, borax; and

may also contain silica and a small amount (0 to 1%) of silver nitrate.

It is important that the lid 22 engages sealingly with the opening to the bottle 20 to ensure that the sodium hydroxide (NaOH) which is corrosive and hygroscopic does not come into contact with the atmosphere during transportation and storage of the bottle and flux.

Each bottle contains a predetermined amount and constitution of flux. The amount of flux, which is usually about 200g, depends on the amount of sample being assayed. Generally, there is a ratio of flux to sample of 4:1 to 6:1, by weight. The constitution of the flux depends on the characteristics of the sample being assayed. For example, a higher amount of borax is added for samples containing higher concentrations of base metals and a higher amount of sodium hydroxide is added for samples containing high amounts of silicates.

An aliquote sample is added to a bottle 20 containing flux 26, the lid is applied back onto the bottle and the sample and flux is mixed by merely shaking the bottle. Previously, mixing containers were used which caused spillages and contamination between samples.

The bottle, flux and sample are then added to an induction furnace 44 as shown in Figure 3. The induction furnace 44 comprises a graphite crucible

46 which is made from soft grain graphite. The crucible 46 is surrounded by a barrier 47 of ceramic material (1600 heat isolating) which in turn is surrounded by an induction coil 48. The aforementioned components are housed in a housing 50 which is made from a ceramic material (polyester board GPO3). The furnace 44 also has a lid 52 which has a backing 54 also made from the ceramic material and an underside 56, made from alumina based ceramic material, which engages sealingly with the opening 58 into the crucible 46, when the lid 52 is closed. It is important that the lid 52 seals the crucible 46, to create a reducing atmosphere therein so that the graphite is not oxidised at high temperatures within the furnace.

The advantage of the flux 26 over known flux compositions is that the sodium hydroxide has a melting point of 318°C, which is much lower than the melting point of CaCO<sub>3</sub>, used in conventional fire assay processes. Thus, when the flux 26 is added to the crucible 46 in the induction furnace 46 (which is normally preheated to a temperature of about 900°C), the sodium hydroxide melts, wets the sample and flux, and retards spattering and boiling when power is applied to the induction furnace. The sodium hydroxide then reacts with silicates in the sample to form slag. Up to now it has not been possible to use sodium hydroxide in conventional fire assay processes as these processes make use of clay pots and the sodium hydroxide would react with silica in the pots and destroy them during heating. The sodium hydroxide does not however have this effect on the graphite crucible 46.

When the sample and flux 26 of the invention are heated in the graphite crucible in the induction furnace, gold and PGMs within the sample are collected within the lead which is stirred vigorously by electromagnetic waves from the induction coil. Because of the speed with which the induction furnace is heated and stirring of the lead, any gold or PGMs in the

sample is collected by the molten lead within 60 seconds of power being applied to the furnace.

Thus, the flux 26, comprising sodium hydroxide makes it possible to rapidly melt an ore sample in an induction furnace as described above without losing flux and ore sample in the process.

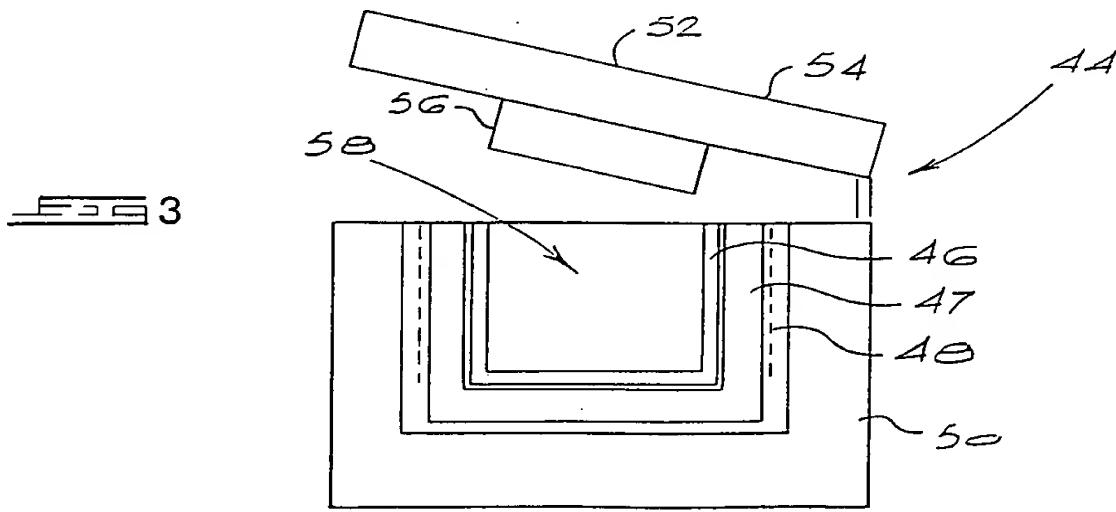
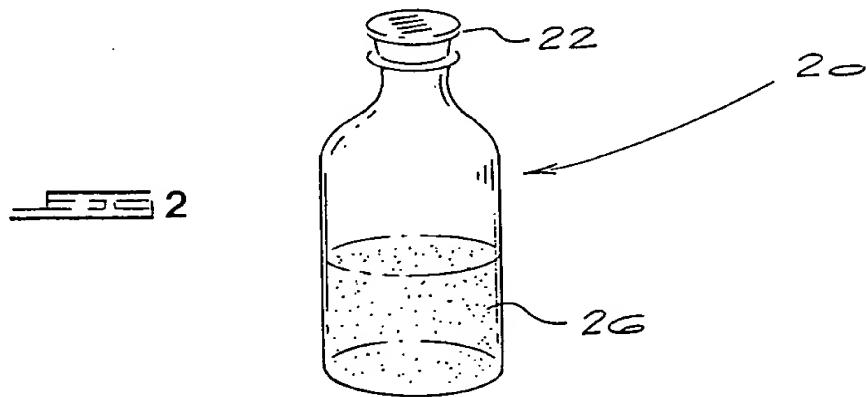
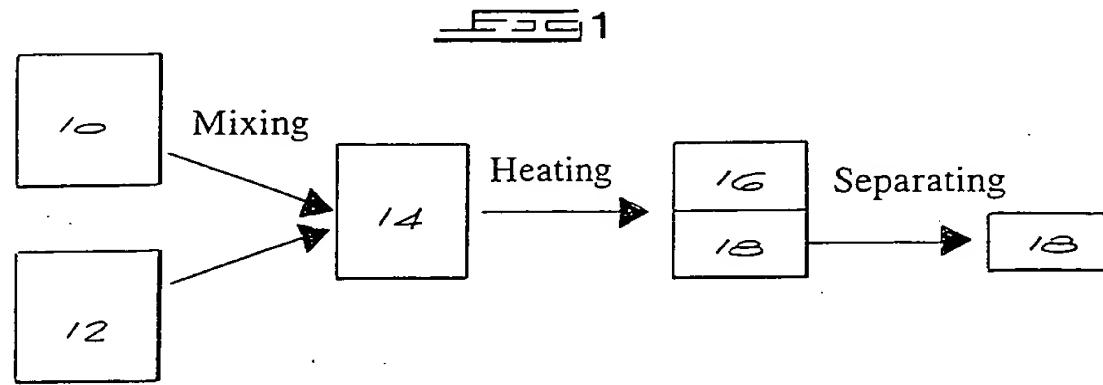
Lastly, the plastics bottle 20 is an important part of the flux composition as it provides a carbon source for reducing the lead oxide to produce molten lead. The graphite crucible also provides a source of carbon.

DATED THIS 29<sup>TH</sup> DAY OF OCTOBER 1998



SPOOR AND FISHER  
APPLICANTS PATENT ATTORNEYS

## PROVISIONAL SPECIFICATION



*Jeff Brown*